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**APPLICATION OF ION IMPLANTATION TO WEAR
PROTECTION OF MATERIALS**



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APPLICATION OF ION IMPLANTATION TO WEAR PROTECTION OF MATERIALS

F.A. Smidt

Ion implantation has many conceptual features that recommend it for use in wear protection applications. The nearly unlimited choice of elemental species which can be implanted, the freedom from thermodynamic constraints, and the ability to perform the implants at low temperatures provide a flexibility in choice of treatment, processing variables and substrate material which cannot be matched by conventional diffusion or coating methods. The combination of ion implantation with processing in a reactive atmosphere extends the possibilities further by utilizing surface reactions and cascade mixing to form surface layers with unique tribological properties. Tribological applications were among the first applications considered for ion implantation in non-semiconductor materials and many glowing reports of success appeared in the literature (1-3). As the number of investigations increased, cases also began to appear where marginal improvements in performance and lack of reproducibility were also observed (4). It is therefore important to establish in a systematic manner the conditions under which ion implantation can be expected to provide improvements in tribological performance, those where it cannot and those where additional research is required.

One of the major problems in defining the conditions under which ion implantation will improve the wear resistance of materials is the complexity of the wear process itself. Systems analysis of the field such as that of Czichos (5) illustrate this point. Factors such as materials in the wear couple, load, configuration, relative sliding velocity, lubrication, environment, and temperature have been identified as major parameters important in the definition of any wear system. Laboratory tests for wear performance tend to reflect the almost infinite combination of the above parameters which might be explored and the statement is often made that the only reliable prediction of wear performance is the actual operation of a system. However, substantial progress has been made in recent years in understanding the response of materials subjected to wear and this provides a partial framework to evaluate the ion implantation results.

Several conferences and reviews provide a good update on recent advances in the field of tribology (6-8). Of particular importance for ion implantation is the recognition that many tribological properties are controlled by thin films adsorbed on the surfaces or at most only a few layers deep (9). Thus surface reactions with the environment and segregation of trace elements to the surface can have a profound effect on friction and wear. Modern surface analysis must be a part of any wear experiment intended to be definitive. Another substantial advance in our understanding has come from the recognition that wear of ductile metals is basically a fatigue process and therefore subject to failure by progressive work hardening, crack nucleation and crack propagation (10,11). This identification of the failure process with fatigue has served to blur the distinctions between the more traditional mechanisms of adhesive and abrasive wear.

A qualitative model of wear therefore includes the following elements: a) initial contacts between the surfaces takes place at asperities which plastically deform to redistribute the load, b) after initial smoothing of the asperities the coefficient of friction is determined by adsorbed species and environmental reactions at the surface, c) the stress transmitted to subsurface region is controlled by the coefficient of friction, the geometry of the contact, and the applied load, d) the rate of accumulation of fatigue damage and failure depends on the material properties, the stress level and the number of fatigue cycles (sliding velocity, distance). e) Temperature of the contact zone is influenced by the coefficient of friction, sliding velocity and applied load. Temperature can influence the rate of accumulation of fatigue damage, metallurgical reactions in the subsurface region and reactions taking place at the surface. At high temperatures a transition to oxidation dominated wear may occur. f) Final failure results from loss of integrity of the surface by delamination and the accumulation of wear debris.

The preceding discussions serve to define the type of test data required to determine the conditions under which implantation provides an improvement in tribological performance. These include a reliable analysis of the surface composition, measurement of the coefficient of friction, well defined test geometry to permit calculation of a Hertzian contact stress, a sliding velocity low enough to disregard temperature excursions and a measure of surface degradation. Tests satisfying the above conditions have been conducted at NRL and Sandia on martensitic steels implanted with reactive ions such as Ti and Ta. These implants form amorphous Fe-Ti(or Ta)-C layers either by dual implantation with C or by surface reactions with adsorbed carbon bearing molecules (12,13) which lower the coefficient of friction. A variety of wear tests have been conducted on these materials and are reviewed in this paper to provide the necessary basis for comparison.

Selected wear tests results which provide information on the benefits of Ti or Ta implants into steels have been analyzed to better define conditions under which wear improvement can be expected. Representative compositions and mechanical properties for these materials are listed in Table 1. A summary of test conditions and observations is provided in table 2.

I. Singer has provided a thorough characterization of surface composition of a variety of steels implanted at NRL with active elements such as Ti (or Ta) (12). Auger analysis has shown that carbon bearing materials in the vacuum chamber chemisorb on the Ti exposed at the surface by sputtering, diffuse inward from the surface at a rate enhanced by the high defect concentrations produced by implantation and eventually form an amorphous Fe-Ti-C layer with a composition ratio of roughly 30 At% Ti, 15 At% C and 55 At% Fe which is about 50 nm thick for an implantation at 190 keV to 5×10^{17} ions/cm². Composition-depth profiles are of course, dependent on ion energy, total fluence and sputtering yield. Most NRL wear experiments have been performed on specimens which are at or near steady state conditions in which the maximum implant concentration with sputtering has been attained and the

original Gaussian profile is distorted to a nearly flat Ti concentration from the surface to the depth of the ion mean range. Auger line shape analysis has shown carbide-like Ti-C bonds in the implanted region.

Wear experiments using a ball on flat geometry have been utilized by Singer (12) to measure changes in the coefficient of sliding friction for a 0.5 in. diameter ball sliding on an implanted flat plate under a 1 Kg load. Wear was measured on the plate as a function of the number of passes using interference microscopy or profilometry traces to measure the depth of the wear scar. Materials examined have included 52100, M-2 and 440 C. A variety of processing conditions have been studied for 52100. These test conditions apply an initial Hertzian contact stress of 830 MPa (120ksi) with a sliding velocity of 0.1 mm/sec (0.24 in/min). Unimplanted 52100 on 52100 shows a coefficient of friction of 0.6 and produces scarring after 10 passes. Implantation of Ti or Ta reduces the coefficient of friction to 0.3 and in specimens which have attained steady state it remains at 0.3 for over 20 passes and no wear scar is observed.

Pope et.al. (14) at Sandia National Laboratories have published a series of papers on the characterization and wear performance of implanted wear materials. They showed independently, that implantation of ferrous alloys with titanium produces an amorphous Fe-Ti-C layer that has low friction and deforms in the wear track. Sandia implantations have used a multiple energy Ti implant with a subsequent C implant to produce concentration profiles with approximately 20 at.% Ti-20 at.%C and 60 at.% Fe to a depth of about 75 nm. This requires a total fluence of 2×10^{17} Ti and C ions/cm². Pin on disk tests have been conducted under unlubricated sliding conditions on a variety of materials including 304 S.S., 15-5 PH and 440 C. Unimplanted test pins with a 0.79 mm tip radius were run against an implanted disk with loads up to 1 Kg. at a sliding velocity of 17 mm/sec (39 in/min). The coefficient of friction was measured during the test and the maximum wear depth determined from a profilometer trace after 1000 cycles was used as a measure of wear. Of particular interest to the present discussion was a set of test results on 440 C with a 440 C pin comparing unimplanted, N implanted and Ti+C implanted surfaces. The unimplanted surface exhibited a coefficient of friction of 0.65 at loads from 50 gr. to 1000 gr. The N implanted specimen initially had a coefficient of friction of .55 and intersected the control curve at about 600 gr. load. Wear of the unimplanted and N implanted specimens increased continuously with load with slightly less wear in the N implanted specimens. The Ti+C implanted specimens by contrast showed a coefficient of friction of 0.3 which remained at that value up to 600 gr., at which point scar depth increased but remained below that of the control up to loads of 1 kg. The load of 600 gr. thus appears to be a threshold value for the onset of wear. This corresponds to an initial Hertzian contact stress of 2800 MPa (408 ksi) which is 1.5 times the uniaxial yield stress. Comparable wear occurs in the unimplanted sample at a contact stress of 1225 MPa (177 ksi). Some blunting of the pin occurs during the pin on disk test so the stress decreases somewhat as the test geometry changes. The Sandia tests are significant in showing a large increase in the stress at which wear is initiated, the relative ineffectiveness of nitrogen implants and the correlation between friction and wear.

Wear tests using the Falex tester were performed on a variety of ion implanted materials by Hartley and Hirvonen at NRL. (15) The Falex test which is used to evaluate lubricants, employs a 0.25 in diameter pin loaded in opposing V blocks. The coefficient of friction is monitored as a function of time during the loading cycle and onset of scuffing wear is manifested by large excursions in the coefficient of friction. The test sequence involved an initial 5 min. run-in period at 250 lb load followed by an increase in load to 700 lbs for 15 min. All tests were run in Hercolube A, a synthetic lubricant based on penterthritol tetrahexanoate. Materials tested included AISI 9310 and 52100 implanted with Ti ions to a fluence of 4.6×10^{17} ions/cm² and Ta ions to a fluence of 1×10^{17} ions/cm². The specimens were implanted by rotating the pins in the beam with an angle of incidence varying from 0-90°. Subsequent analysis of this implantation geometry for Ti has shown the retained dose to be 30-40% of that obtained at normal incidence. Control specimens of both materials showed a coefficient of friction of about .09 with stick-slip peaks of at least twice that value. Ta implantation of 9310 eliminated the stick-slip behavior and a profilometer trace at the end of the test showed an essentially unworn surface compared to a 12 m deep scar in the unimplanted material. A Ti implant of 52100 showed a coefficient of friction of 0.07 vs. 0.09 for the control and showed no stick-slip behavior. Ta implants of 52100 did not show as favorable a performance as on the 9310 and were roughly comparable to the unimplanted material. Calculations of the Hertzian stress for a line contact against four surfaces yielded a value of 890 MPa (130 ksi) for the 700 lb. load. The 290 rpm rotation of the pin gave a sliding velocity of 96 mm/sec (228 in/min). Temperature rises of 180°C were observed during the tests.

Another type of wear test performed on implanted steels is the Faville-6 test in which a ball slider is loaded against a rotating thrust washer under lubricated conditions. The test can be run under sliding velocities in the range from 10-100mm/sec (24-240 in/min.) and under a range of loads. Ramalingam (16) has performed a number of these tests on M-2 steel implanted with Ti under several conditions known to produce a wear resistant layer. Two series of tests were performed. The first involved a 2 hr. test with a failure criteria of an increase in friction during the test period. The second series went to substantially higher loads, monitored changes in friction during a 5 min. test and measured wear behavior with a profilometer trace after the test. All were performed with a mineral oil lubricant and a 52100 ball as the slider. Wear transitions (friction changes) in the 2 hr. low load test were noted at 815 MPa (118 ksi) in the unimplanted specimen and at 1625 MPa (236 ksi) in the specimen implanted to 3.5×10^{17} ions/cm² at 55 keV. A somewhat lower threshold of 1345 MPa (195 ksi) was observed for an implant of 7×10^{17} ions/cm² at 190 keV. The high load tests were somewhat inconclusive in that no friction excursion was noted for the control sample and the 55 keV implant for contact stresses up to 5890 MPa (845 ksi). The 190 keV implant showed a wear transition at 2Kg or a contact stress of 4270 MPa (620 ksi). The inconclusive results in the high load tests were apparently due to the greater wear resistance of M-2 as compared with the 52100 ball since the ball developed a scar during the test. The 190 keV implant may have had a rougher surface due to

sputter relief of the carbides and the implant may also have softened the carbides by amorphization of the surface layer.

Wear tests for specific applications attempt to duplicate the conditions of service as closely as possible. One such test used to evaluate performance of rolling element bearings and bearing lubricants is the geared roller test (17). Two crowned rollers, 3 in. in diameter, are driven independently by gears so that specific amounts of slip take place between the two rollers. Tests have been performed on M50 steel implanted with Ta to a fluence of 1×10^{17} Ta/cm² at 150 keV. The tests were performed at NAPC under the direction of D. Popgoshev with a Hertzian contact stress of 1280 MPa (185 ksi), oil jet lubrication, 1750 rpm and relative slip of 0,7 and 14%. Relative slip is defined as the ratio of the sliding velocity to the mean velocity ((roll + slip)-2) and therefore sliding velocities of 0, 253 and 525 mm/sec. (0, 600, and 1240 in/min) were experienced during the test. The failure mode exhibited in this test is an early form of scuffing wear in which pitting or spallation occurs. Pitting was observed during the 100 hr test duration (10.5 million revolutions) in the unimplanted specimen at 7 and 14% slip. Pitting was observed at 14% slip in the Ta implanted specimen but not at 7% slip.

Another widely used wear test which simulates the service conditions found in gear wear is the Ryder gear test. It is used primarily to evaluate lubricant performance but can also be used to evaluate coatings and surface treatments. The test procedure and data evaluation methods are described in ASTM test D1947. High precision 28 tooth test gears with a 3.5 in. pitch diameter are machined from 9310 steel case hardened and ground. The test is run in a lubricant at 10,000 rpm. A load is applied hydraulically to the gears and a test of 10 min. duration is conducted. The gear teeth are then measured for area of scuffing, the load is increased and the test sequence repeated. The test gear is wider than the drive gears so that it can be reversed and a second set of data obtained on the same gear. The test data are plotted on semi-log paper as percent of tooth area scuffed vs load. A failure criteria of 22.5% tooth area scuffed is applied to the data to obtain a failure load. Ryder gear tests of Ta implanted 9310 were performed at NAPC under the direction of D. Popgoshev and an increase in failure load of 30% was observed relative to an unimplanted gear (18). Contact stresses and sliding velocities for the test geometry were calculated from formulas in the Gear Handbook (19). A contact stress of 1240 MPa (180 ksi) was calculated for the control gear and a stress of 1420 MPa (206 ksi) was calculated for the Ta implanted gear. Sliding velocity on the gear tooth varied from 0 at the pitch diameter to a maximum of 14,700 mm/sec (35,000 in/min) at the gear tip.

Metal cutting operations have also been examined as a service test of Ti implanted M-2 tool steel (20). Ramalingam measured cutting forces in an instrumented lathe while cutting annealed 4340 steel. A reduction in Flank wear by a factor of two was observed on duplicate tests and a reduction in power requirements of about 10% was also observed. A calculation of sliding velocity of the metal chip relative to the tool face yielded a value of 570mm/sec (1370 in/min). An analysis of the stresses exerted on the tool face was performed using the tool force

equations cited by Shaw (21) and Wright (22) and assuming a rake angle of 60° as the best fit of the force components to a shear stress of 435 MPa for annealed 4340. The normal stress exerted on the tool cutting edge was estimated to be in the range 290-385 MPa (42-56 ksi) assuming a cutting length of 1mm to .75 mm respectively. Wear under metal cutting conditions is strongly influenced by heating of the tool to temperatures in excess of 650°C and abrasive cutting of the flank by hard inclusions in the workpiece.

Discussion

The comparison of test results from a variety of wear tests of commercial wear resistant steels which have been implanted with reactive species known to react with carbon in the near surface region helps to define the conditions under which this treatment can improve wear performance. Two additional pieces of information are useful in interpreting these results, the force-velocity dependence of wear in lubricated systems and the wear behavior of surfaces with hard coats.

Begelinger and DeGee (23) have developed a wear transition diagram to describe the wear behavior of highly loaded lubricated contacts. At low loads the surfaces are separated by elasto hydrodynamic films (EHD) (region I) and friction of the system is low ($\mu=0.07$ or less). At higher loads, a transition to boundary lubrication occurs (region II) with an initially high coefficient of friction ($\mu=.35$) as asperities make contact through the film; the friction and wear rate decrease ($\mu=.12$) as the asperities are rounded or oxidized by localized frictional heating. At still higher loads and velocities metal to metal contact occurs, the coefficient of friction rises to 0.35 - 0.5 and severe scuffing wear (region III) ensues. The transition from I to III takes place directly for sliding velocities above about 1m/sec for steels. This test procedure has been applied to the evaluation of CVD hard coats of TiC, TiN and $(\text{Cr, Fe})_7\text{C}_3$ applied to 52100 steel (24). For tests run at 500mm/sec wear transitions for I-II occurred at 1690 to 2760 MPa (245-400 ksi) depending on surface roughness and for II-III at 3725 MPa (540 ksi). Coating one surface with TiC increased the transitions to 5245 MPa (760ksi) and 5845 MPa (847 ksi) respectively, TiN increased both transitions above 7830 MPa (1135ksi) and $(\text{CrFe})_7\text{C}_3$ increased the transitions only slightly to 5900 MPa (420 ksi) and 3800 MPa (550 ksi). Coating of both surfaces produced less beneficial results as the load-carrying capacity of the partial EHD film is limited by removal of the CVD coating on the ring surface.

Several observations are pertinent before making a detailed analysis of the test data. First, the conditions of the tests which simulate wear under typical service conditions (Geared roller, Ryder gear) tend to be at higher sliding velocities and for longer test periods than the laboratory tests. The failure mode under these conditions is fatigue. Second, operating contact stresses are primarily determined by the level at which EHD films breakdown since long term operation of machinery requires wear rates comparable to those in region I of the Begelinger - DeGee wear transition diagram. Thirdly, high stress-short time tests with a sliding component tend to wear by inducing plastic flow of the surface. Finally, surface treatments primarily influence wear by

modifying the coefficient of friction or by hardening the surface. The effect of surface hardening in reducing wear is well known and has been commercially exploited for many years. The influence of friction on heating has also been recognized for a long time but a more recent advance has been the correlation of the coefficient of friction with the coupling of stress into the region below a contact spot. Suh and his coworkers (10) calculated subsurface stress fields and plastic envelopes for yielding as a function of load and coefficient of friction, showed the maximum stress moved from subsurface to surface for $\mu > 0.3$, and related the calculated stress fields to the nucleation and propagation of subsurface cracks. Singer (25) has applied these concepts to formulate a wear reduction mechanism for Ti ion implanted steels where a reduction in coefficient of friction is observed.

The ion implantation data in table 1 are interpreted in the following discussion. The slow speed unlubricated sliding ball experiments of Singer show a reduction in coefficient of friction due to the Fe-Ti-C amorphous layer and this in turn reduces the shear stress coupled to the surface and increases the threshold for scarring the surface. The Sandia experiments on 440C are the most impressive increase in wear performance demonstrated to date. They appear to combine both a reduced friction mechanism and a hardened surface layer which is thick enough to suppress subsurface deformation. A hardened surface layer has been shown by microhardness measurements to occur when dual implants of Ti and C in a stoichiometric ratio are implanted while a hardness increase is not observed for implantation in a reactive atmosphere (26). The Falex tests performed by Hartley and Hirvonen were above the EHD breakdown limit for lubricated wear of steels and thus demonstrated qualitatively that implantation could increase the threshold for scuffing wear. The mechanism was predominantly friction reduction which reduced the severity of asperity contacts and prevented destruction of the boundary layer. The low load -2 hr. Faville -6 tests conducted by Ramalingam were in the same range of load and velocity but had a less severe point contact rather than a line contact. Roughness of the surface produced by sputter relief of carbides in the M2 appeared to be more important than depth of the implanted layer in influencing the EHD to boundary layer transition. The high-load -5 min. Faville 6 tests appeared to be controlled by the hardness of the M2 carbides. Roughening of the surface or softening of the carbides by amorphization degraded the wear resistance of the 190 keV implant below that of the unimplanted and 55 keV implant specimens. The geared roller test showed implantation delayed the onset of spallation damage from 7% slip to 14% slip probably by a friction reduction mechanism. The Ryder gear test showed an increase in the threshold for scuffing wear by the same mechanism. The metal cutting test, while conducted at relatively modest stress levels, produced an increase in tool temperature not encountered in the other tests which reduced the relative yield stress ratio to about the level where failure occurred by surface deformation in other tests.

Conclusion

Ion implantation can improve the wear resistance of bearing and tool steels by lowering the coefficient of friction of the surface and by producing a thin hard layer on the surface. The latter mechanism is

generally less effective at high loads than a coating laid down by some vapor deposition process although it may provide some benefit in delaying ploughing of the surface by asperity contact through an EHD or boundary layer film. Additional research is needed to determine the optimum thickness for coatings but 1-2 μm appears to be adequate for substantial improvements in wear. Films of this thickness can only be achieved by ion beam enhanced deposition or by repetitive deposition and ion beam mixing. Friction reduction, even in the absence of a hardened surface layer, appears to be effective in raising the threshold for scuffing wear under lubricated conditions by 30-100%. Within this range of stresses, one would expect to see substantial increases in wear life. Additional research is required to understand what benefits are possible under high speed, lubricated sliding wear conditions and the durability of the low friction implanted layer.

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TABLE 1 MATERIALS COMPOSITION AND PROPERTIES

Materials	Composition (wt%)					Max. Operating Temp. °C	Knoop Hardness	Yield Strength	
	C	Cr	Mn	Si	Mo	Other		MPa	ksi
52100	1.05	1.45	.35	.22	-	-	1500	1860	270
M2	.82	3.85	.30	.30	5.0	6.1 W, 1.9V	535	2105	305
440C	1.03	17.3	.48	.41	.48	.14V	230	1840	270
M50	.80	4.0	.27	.17	4.25	1.0V	425	2015	292
9310*	.10	1.2	.55	.22	.12	3.25 Ni	-	905	132

*Case carburized to KHN ≈ 800

TABLE II SUMMARY OF WEAR TESTS ON A VARIETY OF FERRITIC MATERIALS IMPLANTED WITH CARBIDE FORMING SPECIES

Test	Materials Slider Disk	Implant	Lubrication Conditions	Hertzian stresses ksi	Hertzian MPa	Sliding Velocity in/min	Failure Criteria	Observations
1/2" diam ball on flat (NRL)	52100/52100 52100/M-2	- Ti	Unlube Unlube	120 120	830 830	0.24 0.24	depth of scar	$\mu=.6$ Scar after 20 passes, $\mu=.3$ no scar
Pin on disk test (Sandia)	440C/440C 440C/440C	- Ti+C+	Unlube Unlube	177 408	1225 2800	40 40	17 17	deep scar, High Wear Rate onset of severe wear
Faville 6 Series 1 (Ramalingam)	52100/M2 52100/M2	- Ti+	Mineral Oil Mineral Oil	120 236	815 1625	24-240 24-240	increase in friction	load increased at given velocity until increase in observed.
Faville 6 Series 2 (Ramalingam)	52100/M2 52100/M2 52100/M2	- Ti+ hot implant	Mineral Oil Mineral Oil Mineral Oil	845 845 845	5890 5890 5890	27-29 27-29 27-29	wear scar depth	no scar - ball wear mixed results - No scar
Falix (NRL)	52100/52100 9310/9310 52100/52100 9310/9310 52100/52100 9310/9310	- Ti+ Ta+ - Ti, Ta	Herculube A Herculube A Herculube A Herculube A	66 66 130 130	455 455 890 890	228 228 228 228	96 96 96 96	20X lower wear rate for implant Ti+, Ta+ stick-slip, deep scar no stick, very shallow scar
Machining Instrumented Lathe (Ramalingam)	Cut Tool M-Z 4340	- Ti+	Unlube Unlube	*42-56	290 385	370 370	570 570	1/2 wear rate, 10% less power
Geared Roller NAPC, 0.7, 14% slip	M50/M50 M50/M50	- Ta+	Oil Jet Oil Jet	185 185	1280 1280	600 1242	253 525	No wear at 0% pitting at 14% no damage in implant at 7%
Ryder Gear	9310/9310 9310/9310	- Ta+	PESL-1191A PESL-1191A	180 206	1240 1420	Variable 0-15,000	0-3500 scoring	30% increase in load capacity
Rolling Contact Fatigue Variety of tests	M50 & 440C N, Cr, & Ta	- N, Cr, & Ta	None None	700 700	4830 4830	no slip	Surface damage	No Major Change except possibly with Ta

* Applied Stress